

## CHAPTER 3

### DETERMINING INSTRUMENT ACCURACY

Section Number	Section	Page Number
3.1	STANDARD REFERENCE AND SLOPE SAMPLE SETS	3-1
3.2	WHEAT BIAS ADJUSTMENTS	3-6
3.3	SOYBEAN BIAS ADJUSTMENTS	3-12
3.4	CORN BIAS ADJUSTMENTS	3-17
3.5	INSTRUMENT CHECKOUT	3-23
	Attachment 1 - NIRT Daily Wheat SRS Worksheet	
	Attachment 2 - NIRT Daily Soybean SRS Worksheet	
	Attachment 3 - NIRT Weekly Wheat SRS Worksheet	
	Attachment 4 - NIRT Weekly Soybean SRS Worksheet	
	Attachment 5 - NIRT Wheat SRS Bias Log	
	Attachment 6 - NIRT Soybean SRS Bias Log	
	Attachment 7 - NIRT Daily Corn SRS Worksheet	
	Attachment 8 - NIRT Weekly Corn SRS Worksheet	
	Attachment 9 - NIRT Corn SRS Bias Log	



## CHAPTER 3

### DETERMINING INSTRUMENT ACCURACY

#### 3.1 STANDARD REFERENCE AND SLOPE SAMPLES

a. General.

- (1) Standard Reference (SRS) samples consist of bulk quantities of wheat, soybeans, and corn with established protein, oil, and starch values (as applicable) determined by ISE.
- (2) Slope samples are used to adjust the slope of the TSD NIRT instruments to agree optimally with the standard reference methods for each calibration. The average slope and intercept value from the TSD instruments are then used to determine the standard slope settings for each calibration.
- (3) SRS baseline values are corrected to a 12 percent moisture basis (mb) for wheat, 13 percent mb for soybeans, and a dry matter basis for corn before being released with the SRS sets.
- (4) SRS sets are used to test instrument accuracy and to determine the amount of bias adjustments needed. SRS may no longer be used if they weigh less than 500 grams.
- (5) Contact your NIRT coordinator or ISE to request SRS sample sets. The ISE will supply SRS that typically weigh between 650 and 750 grams for wheat, between 550 and 650 grams for soybeans, and between 575 and 675 grams for corn.

- b. Nature and Number of Samples. ISE will select a set of four (4) samples for Corn, and five (5) samples each of Hard Red Winter wheat (HRW), Hard Red Spring wheat (HRS), Soft Red Winter wheat (SRW), Soft White wheat (SWH), Hard White wheat (HDWH), Durum wheat (DU), and Soybeans. ISE will issue a unique baseline value for each class of wheat, for soybean protein and oil, and for corn protein, oil, and starch. Use the proper SRS set and baseline value for the class of wheat being tested. **Perform a duplicate determination on each wheat and corn SRS sample and a single determination on each soybean SRS sample.**

- c. Developing Baseline Values. The wheat protein, soybean protein and oil, and corn protein, oil, and starch baseline values are determined by ISE on the standard NIRT instrument. ISE instruments are directly compared and adjusted to optimally agree with the standard reference method.
- d. Replacing Reference Samples. ISE will maintain bulk reference samples to replace the field supply as they become infested, contaminated, or depleted. Testing locations must replace the wheat SRS set(s) after 6 months. The 6-month period starts on the day the SRS are first used. It should be within 2 weeks of receiving the SRS from ISE; if not, the SRS should be refrigerated until they are ready for use. Soybean and corn SRS sets will be replaced on a yearly basis unless they become infested, contaminated, or depleted. Request a replacement supply from ISE as far in advance as possible (30 days recommended).

Immediately notify your NIRT coordinator or ISE if any of the SRS sample(s) becomes contaminated or infested and obtain replacement(s).

In case of emergencies, SRS can be shipped overnight during the week. Otherwise, the SRS will be shipped through United Parcel Service (UPS) and should arrive within 5 business days of placing an order for replacement samples.

- (1) Collecting and Testing Replacement Samples. ISE will collect, evaluate, and select SRS. ISE will evaluate and select slope sets when calibrations are updated. ISE may request selected testing locations to provide samples for this purpose.
- (2) Replacing SRS or Baseline Values. A smooth transition should occur when new reference sample(s) and/or baseline value(s) are implemented. **This procedure does not apply to any changes associated with implementing a new calibration or standard slope setting.** Before replacing old reference sample set(s) or baseline value(s), perform the following:
  - (a) Check the instrument bias to the **old** baseline values using the old SRS. Record the average difference between the NIRT and baseline.
  - (b) Check, but do not correct, the instrument's bias term using the new SRS samples and baseline values. Record the average difference between the NIRT and baseline.
  - (c) Repeat steps (a) and (b) two additional times within a period not to exceed 2 weeks.

- (d) Review the three data sets. The new reference sample(s) with the new baseline value(s) may be used and the old reference sample(s) with the old baseline value(s) discarded if the amount of bias shown by the new set agrees with the amount of bias shown by the old set within an average of  $\pm 0.05$  percent protein or oil, or  $\pm 0.20$  starch.

Contact your NIRT coordinator or ISE for assistance if the new reference sample(s) do not meet the above requirements. Continue to use the old reference sample(s) for bias adjusting the instrument until the reason for the discrepancy is determined and corrected. If the new reference sample(s) meet the above requirement, they may now be used to check the instrument bias prior to running market samples.

- (3) Replacing SRS or Baseline Value(s) as a Result of Calibration Update or Adjustment. When a calibration update or adjustment and a new baseline are implemented simultaneously, no phase-in is required. Once the standard slope settings are entered in the instrument or on the calibration disk, test the new SRS two times. Use the Level II tolerances to determine if a bias adjustment is needed. Contact ISE with your final intercept values for the updated and/or adjusted calibrations.
- (4) Storage of Standard Reference Samples.
  - (a) Store reference samples at room temperature in plastic containers with screw tops or securely fastened lids and place away from vents, heating devices, and direct sunlight. To prevent insect infestation, operators may place mothballs sealed in plastic bags (with small holes) with the reference samples. The mothballs must remain sealed in the plastic bags at all times. Operators must remove any mothballs before testing the SRS set.
  - (b) SRS samples may be stored under refrigeration. However, the SRS must be allowed to reach room temperature ( $\pm 5^{\circ}\text{F}$ ) before testing.
  - (c) Discard any SRS sample if it becomes infested or contaminated. Immediately inform your NIRT coordinator or ISE and obtain replacement sample(s).

- (d) Protect reference samples from manipulation by unauthorized persons to maintain sample integrity.
- e. Using Standard Reference Samples. Before testing market samples, the operator must first test, evaluate, and, if necessary, bias adjust the instrument using the appropriate SRS set and baseline values for soybeans, corn, or for the class of wheat being tested as follows:
- (1) Mix each SRS several times before testing.
  - (2) Analyze each sample according to the official procedures (see section 4.3.a).
  - (3) Review the NIRT values and apply the appropriate individual, average, and range differences between the NIRT and baseline value(s) (see section 3.1.f for wheat, 3.1.g for soybeans, and 3.1.h for corn).
  - (4) Record all data. Examples of a daily SRS worksheet, a weekly SRS worksheet, and a bias log are given in attachments 1-9. Testing locations may use these attachments or develop a log or worksheet to best suit their needs.
- f. Wheat Data Evaluation. Evaluate the SRS values by comparing individual and average NIRT protein to the corresponding baseline value(s). During data evaluation, if any of the following conditions occur, the operator must take the necessary corrective action before providing official protein testing service:
- (1) Duplicate Difference. If the difference between the first and second analysis of an individual SRS exceeds  $\pm 0.20$  percent protein, then reanalyze the sample. Record the results from the two analyses closest to each other and discard the third result.  
  
Duplicate Difference = an individual sample NIRT value first analysis minus (-) second individual sample NIRT value.
  - (2) Individual Difference. An individual reference sample NIRT protein value differs from its baseline value by **more than**  $\pm 0.40$  percent protein.

The individual difference is calculated as follows:

Individual difference = an individual sample NIRT protein value minus (-) sample baseline value.

- (3) Average Differences. The average difference between the SRS NIRT protein values and the baseline values exceeds the **applicable tolerance level** (see section 3.2 for more information).
- g. Soybean Data Evaluation. Evaluate the SRS values by comparing individual and average NIRT protein and oil to their corresponding baseline value(s). During data evaluation, if any of the following conditions occur, the operator must take the necessary corrective action before providing official protein and oil testing services:
- (1) Individual Difference. An individual reference sample NIRT protein value differs from its baseline value by **more than**  $\pm 0.40$  percent protein. An individual reference sample NIRT oil value differs from its baseline value by **more than**  $\pm 0.30$  percent oil.
- (2) Average Differences. The average difference between the SRS NIRT protein and/or oil values and the baseline values exceeds the **applicable tolerance level** (see section 3.3 for more information).
- h. Corn Data Evaluation. Evaluate the SRS values by comparing individual and average NIRT protein, oil, and starch to their corresponding baseline value(s). During data evaluation, if any of the following conditions occur, the operator must take the necessary corrective action before providing official protein, oil, and starch testing services:
- (1) Duplicate Difference. If the difference between the first and second analysis of an individual SRS exceeds  $\pm 0.30$  percent protein,  $\pm 0.40$  percent oil, or  $\pm 0.90$  percent starch, then reanalyze the sample for the constituent of interest. Record the results from the two analyses closest to each other for the constituent of interest and discard the third result.
- Duplicate Difference = an individual sample NIRT value first analysis minus (-) second individual sample NIRT value.
- (2) Individual Difference. An individual reference sample NIRT protein value differs from its baseline value by **more than**  $\pm 0.40$  percent protein. An individual reference sample NIRT oil value differs from its baseline value by **more than**  $\pm 0.50$  percent oil. An individual reference sample NIRT starch value differs from its baseline value by **more than**  $\pm 0.80$  percent starch.

- (3) Average Differences. The average difference between the SRS NIRT protein and/or oil and/or starch values and the baseline values exceeds the **applicable tolerance level** (see section 3.4 for more information).

## 3.2 WHEAT BIAS ADJUSTMENTS

### a. General.

- (1) The NIRT instrument may, at times, either read high or low when compared to the SRS baseline values. Systematic deviation from the baseline values greater than the allowable tolerance limits must be corrected before official wheat protein testing services are performed.
- (2) All NIRT instruments are equipped with an "intercept" constant which is used to adjust the instrument's bias with respect to the baseline. If the intercept constant is set properly, the instrument should give results which, on average, are accurate with respect to ISE standard NIRT instruments. Every effort shall be made to keep the calculated average bias of all NIRT instruments within  $\pm 0.05$  percent protein at all times. Adjust the bias of the NIRT calibration by subtracting the calculated average difference between the SRS results and the baseline value from the instrument's INTERCEPT constant for the calibration. This may mean running extra sets of SRS to have two or three valid sets available.
- (3) Prior to official wheat protein testing, perform a bias check and, if necessary, adjust the intercept constant for each wheat class being tested.
- (4) Determine instrument accuracy by comparing SRS results to their established baseline values.
- (5) Only SRS results obtained using procedures identical to market sample procedures should be used in computing bias adjustments.
- (6) Any change in bias (especially if the bias has been stable for some time) may be an indication of procedural or equipment problems. If in doubt, check for possible problems before making an adjustment.
- (7) It is permissible to repeat the entire SRS set and discard prior SRS results if the results appear to be in error.
- (8) Maintain bias records at each location at all times.



- b. Tolerance Levels. In addition to the individual sample difference limit of  $\pm 0.40$  percent protein, four average difference tolerance levels were developed. These tolerance levels allow testing locations to bias to tighter tolerances. The applicable tolerance level is based on the number of valid SRS data sets available.

Presently, the four tolerance levels are set as follows:

(1) **LEVEL-I ( $\pm 0.10$  percent protein tolerance):**

Apply a LEVEL-I tolerance when evaluating a single valid SRS data set (typically an average of 10 results).

(2) **LEVEL-II ( $\pm 0.07$  percent protein tolerance):**

Apply a LEVEL-II tolerance when evaluating the average of two valid SRS data sets (typically an average of 20 results) collected within a 2-week period and within a 5°F temperature range.

(3) **LEVEL-III ( $\pm 0.05$  percent protein tolerance):**

Apply a LEVEL-III tolerance when evaluating the average of three consecutive SRS data sets (typically an average of 30 results) collected within a 2-week period and within a 5°F temperature range.

(4) **LEVEL-IV ( $\pm 0.03$  percent protein tolerance):**

Apply a LEVEL-IV tolerance when evaluating the average of five valid SRS data sets (typically an average of 50 results) that are all positive or negative and were collected within a 2-week period and within a 5°F temperature range. An average difference between a single valid SRS data set and the baseline of zero is neither positive or negative.

Record all bias adjustment data in the maintenance log and on the SRS log and/or worksheets (see attachments for examples). Include the date of the bias adjustment, the suspected source of error (if known), any action taken to correct the problem, direction provided by the NIRT coordinator or ISE, and the magnitude of the adjustment. All locations must submit a copy of all SRS information recorded during the week to the monitoring field office. The field office manager may request SRS information more frequently if a problem is suspected.

- c. Performing a Bias Check or Adjustment. Previous SRS NIRT results are invalidated after a change in the standard slope, bias adjustment, instrument repair or replacement, or when the recorded temperature has varied by more than  $\pm 5^{\circ}\text{F}$ .

Perform a bias check: (1) once a day when the instrument is turned on and warmed up, or after power is restored after a power outage; (2) the instruments accuracy is questioned; (3) the RH is outside of the acceptable range or RH returns to the acceptable range and a bias adjustment was made while the RH was outside the acceptable range; or (4) the temperature changes by more than  $\pm 5^{\circ}\text{F}$  from the temperature recorded during the daily check.

**Note: After changing between the 18 millimeter and 30 millimeter sample cell, select the high and low protein samples from one of the wheat SRS. Run these samples as a check to verify that the sample cell is installed correctly. The samples should yield results similar to the results made prior to changing the sample cell. If not, reinstall the sample cell making sure the area is clear of any obstructions.**

The following procedure outlines the instrument check and evaluation of two kinds of data. These data concern (1) individual differences from the baseline and (2) the data related to the average differences from the baseline. Each is used to determine the relative accuracy of the equipment and the amount of correction (biasing) needed to allow the equipment to duplicate the values of the known SRS baseline values. The instrument check and data evaluation procedure start with "STEP 1" then proceed through a series of "YES" and "NO" responses to questions until being directed to analyze market samples.

**STEP 1:** Mix each SRS thoroughly before analyzing.

**STEP 2:** Calculate the difference between the duplicate analyses for the same sample. Does the duplicate difference for any sample differ by more than  $\pm 0.20$  percent protein?

- a. If **NO**, proceed to STEP 3.
- b. If **YES**, reanalyze the sample. Record and use the results from the two analyses closest to each other and discard the third result. Proceed to STEP 3.

**STEP 3:** Calculate individual analysis differences between the NIRT and baseline. Does any SRS differ by more than  $\pm 0.40$  percent protein from its baseline value?

- a. If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
- b. If **YES**, calculate the range of difference between the NIRT and baseline. Is the range greater than 0.50 percent protein?

The range difference is found by identifying the most positive individual difference and identifying the most negative individual difference then calculating the difference between the two extreme values. See the following examples for more information:

Example 1. Where positive and negative differences are observed:

If the largest positive difference is + 0.34 and the largest negative difference is - 0.39, then the range difference is  $(+ 0.34) - (- 0.39) = + 0.73$  percent protein.

Example 2. Where all observed differences are positive:

If the largest positive difference is + 0.34 and the smallest positive difference is +0.09, then the range difference is  $(+ 0.34) - (+ 0.09) = + 0.25$  percent protein.

Example 3. Where all observed differences are negative:

If the smallest negative difference is - 0.09 and the largest negative difference is -0.39, then the range difference is  $(- 0.09) - (- 0.39) = + 0.30$  percent protein.

- (1) If **YES**, reanalyze deviating sample(s). Drop from the average any sample that remains more  $\pm 0.40$  percent protein different from its baseline value and notify your NIRT coordinator. Calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
- (2) If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.

**STEP 4:** Review available data sets. (Data sets older than 2 weeks or intervening bias adjustment, new standard slope, instrument repairs, etc., render SRS data invalid).

- a. Determine whether the average difference between the NIRT and baseline is  $\pm 0.10$  percent protein.
  - (1) If the average difference between the NIRT and baseline is  $\pm 0.10$  percent protein or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.b.
  - (2) If the average difference between the NIRT and baseline is greater than  $\pm 0.10$  percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
    - (a) If the corrected difference is  $\pm 0.05$  percent protein or less, proceed to analyze market samples.
    - (b) If the difference is still greater than  $\pm 0.05$  percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- b. If the previous data set is valid, calculate the average difference from the baseline for the two sets (20 individual analyses).
  - (1) If the average difference is  $\pm 0.07$  percent protein or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.c.
  - (2) If the average difference is greater than  $\pm 0.07$  percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
    - (a) If the corrected difference is  $\pm 0.05$  percent protein or less, proceed to analyze market samples.
    - (b) If the difference is still greater than  $\pm 0.05$  percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- c. If the previous three data sets are valid, calculate the average difference from the baseline for the three sets (30 individual analyses).

- (1) If the average difference is  $\pm 0.05$  percent protein or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.d.
  - (2) If the average difference is greater than  $\pm 0.05$  percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
    - (a) If the corrected difference is  $\pm 0.05$  percent protein or less, proceed to analyze market samples.
    - (b) If the difference is still greater than  $\pm 0.05$  percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- d. If the previous data sets are valid, check the last five runs to see if they are all positive or negative, excluding any zeroes. If they are all positive or negative, then calculate the average difference from the baseline for five sets (50 individual analyses), otherwise proceed to analyze market samples.
- (1) If the average difference is  $\pm 0.03$  percent protein or less, proceed to analyze market samples.
  - (2) If the average difference is greater than  $\pm 0.03$  percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
    - (a) If the corrected difference is  $\pm 0.05$  percent protein or less, proceed to analyze market samples.
    - (b) If the difference is still greater than  $\pm 0.05$  percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.

### 3.3 SOYBEAN BIAS ADJUSTMENTS

#### a. General.

- (1) The NIRT instrument may, at times, read either high or low when compared to the SRS baseline values. Systematic deviation from the baseline values greater than the allowable tolerance limits must be corrected before official soybean protein and oil testing services are performed.
- (2) All NIRT instruments are equipped with an "intercept" constant which is used to adjust the instrument's bias with respect to the baseline. If the intercept constant is set properly, the instrument should give results which, on average, are accurate with respect to ISE standard NIRT instruments. Every effort should be made to keep the calculated average bias of all NIRT instruments within  $\pm 0.08$  percent protein and  $\pm 0.05$  percent oil at all times.

Adjust the bias of the NIRT calibration by subtracting the calculated average difference between the SRS results and the baseline value from the instrument's INTERCEPT constant for the constituent of interest. This may mean running extra sets of SRS to have two or three valid sets available.

- (3) Prior to official soybean protein and oil testing, perform a bias check and, if necessary, adjust the intercept constant for each constituent.
- (4) Determine instrument accuracy by comparing SRS results to their established baseline values.
- (5) Only SRS results obtained using procedures identical to market sample procedures should be used in computing bias adjustments.
- (6) Any change in bias (especially if the bias has been stable for some time) may be an indication of procedural or equipment problems. If in doubt, check for possible problems before making an adjustment.
- (7) It is permissible to repeat the entire SRS set and discard prior SRS results if the results appear to be in error.
- (8) Maintain bias records at each location at all times.

- b. Tolerance Levels. In addition to the individual sample difference limit of  $\pm 0.40$  percent protein and  $\pm 0.30$  percent oil, four average difference tolerance levels were developed. These tolerance levels allow testing locations to bias to tighter tolerances. The applicable tolerance level is based on the number of valid SRS data sets available.

Presently, the four tolerance levels are set as follows:

(1) **LEVEL-I ( $\pm 0.17$  percent protein and  $\pm 0.12$  percent oil tolerances):**

Apply a LEVEL-I tolerance when evaluating a single valid SRS data set (typically an average of 5 results).

(2) **LEVEL-II ( $\pm 0.12$  percent protein and  $\pm 0.09$  tolerances):**

Apply a LEVEL-II tolerance when evaluating the average of two valid SRS data sets (typically an average of 10 results) collected within a 2-week period and within a 5°F temperature range.

(3) **LEVEL-III ( $\pm 0.10$  percent protein and  $\pm 0.07$  percent oil tolerances):**

Apply a LEVEL-III tolerance when evaluating the average of three consecutive SRS data sets (typically an average of 15 results) collected within a 2-week period and within a 5°F temperature range.

(4) **LEVEL-IV ( $\pm 0.08$  percent protein and  $\pm 0.05$  percent oil tolerances):**

Apply a LEVEL-IV tolerance when evaluating the average of five valid SRS data sets (typically an average of 25 results) that are all positive or negative and were collected within a 2-week period and within a 5°F temperature range. An average difference between a single valid SRS data set and the baseline of zero is neither positive or negative.

- c. Performing a Bias Check or Adjustment. Previous SRS NIRT results are invalidated after a change in the standard slope, bias adjustment, instrument repair or replacement, or when the recorded temperature has varied by more than  $\pm 5^\circ\text{F}$ .

Perform a bias check when: (1) once a day when the instrument is turned on and warmed up, or after power is restored after a power outage; (2) the instrument's accuracy is questioned; (3) the RH is outside of the acceptable range or RH returns to the acceptable range and a bias adjustment was made while the RH was outside the acceptable range; or (4) the temperature changes by more than  $\pm 5^{\circ}\text{F}$  from the temperature recorded during the daily check.

**Note: After changing between the 18 millimeter and 30 millimeter sample cell, select the high and low protein SRS. Run these samples as a check to verify that the sample cell is installed correctly. The samples should yield protein results similar to the results made prior to changing the sample cell. If not, reinstall the sample cell making sure the area is clear of any obstructions.**

The following procedure outlines the instrument check and evaluation of two kinds of data. These data concern (1) individual differences from the baseline and (2) the data related to the average differences from the baseline. Each is used to determine the relative accuracy of the equipment and the amount of correction (biasing) needed to allow the equipment to duplicate the values of the known SRS baseline values. The instrument check and data evaluation procedure start with "STEP 1" then proceed through a series of "YES" and "NO" responses to questions until being directed to analyze market samples.

**STEP 1:** Mix each SRS thoroughly before analyzing.

**STEP 2:** Calculate individual sample differences between the NIRT and baseline. Do any SRS differ by more than  $\pm 0.40$  percent protein or  $\pm 0.30$  percent oil from its baseline value?

- a. If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 3.
- b. If **YES**, calculate the range of difference between the NIRT and baseline. Is the range greater than 0.60 percent protein or 0.45 percent oil?

Examples for calculating the range difference between the two extreme values can be found on page 3-8.

- (1) If **YES**, reanalyze deviating sample(s). Drop from the average any sample that remains more  $\pm 0.40$  percent protein or  $\pm 0.30$  percent oil different from its baseline value and notify your NIRT coordinator. Only one sample can be dropped from the average. If more than one sample exceeds the tolerance, contact ISE.



Calculate the average difference between the NIRT values and baseline. Proceed to STEP 3.

- (2) If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 3.

**STEP 3:** Review available data sets. (Data sets older than 2 weeks or intervening bias adjustment, new standard slope, instrument repairs, etc., render SRS data invalid).

- a. Determine whether the average difference between the NIRT and baseline is  $\pm 0.17$  percent protein or  $\pm 0.12$  percent oil.
  - (1) If the average difference between the NIRT and baseline is  $\pm 0.17$  percent protein or less and  $\pm 0.12$  percent oil or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 3.b.
  - (2) If the average difference between the NIRT and baseline is greater than  $\pm 0.17$  percent protein or  $\pm 0.12$  percent oil, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
    - (a) If the corrected difference is  $\pm 0.08$  percent protein or less or  $\pm 0.05$  percent oil or less, proceed to analyze market samples.
    - (b) If the difference is still greater than  $\pm 0.08$  percent protein or  $\pm 0.05$  percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- b. If the previous data set is valid, calculate the average difference from the baseline for the two sets (10 individual analyses).
  - (1) If the average difference is  $\pm 0.12$  percent protein or less and  $\pm 0.09$  percent oil or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 3.c.

- (2) If the average difference is greater than  $\pm 0.12$  percent protein or  $\pm 0.09$  percent oil, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
  - (a) If the corrected difference is  $\pm 0.08$  percent protein or less or  $\pm 0.05$  percent oil or less, proceed to analyze market samples.
  - (b) If the difference is still greater than  $\pm 0.08$  percent protein or  $\pm 0.05$  percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- c. If the previous three data sets are valid, calculate the average difference from the baseline for the three sets (15 individual analyses).
  - (1) If the average difference is  $\pm 0.10$  percent protein or less and  $\pm 0.07$  percent oil or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 3.d.
  - (2) If the average difference is greater than  $\pm 0.10$  percent protein or  $\pm 0.07$  percent oil, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
    - (a) If the corrected difference is  $\pm 0.08$  percent protein or less or  $\pm 0.05$  percent oil or less, proceed to analyze market samples.
    - (b) If the difference is still greater than  $\pm 0.08$  percent protein or  $\pm 0.05$  percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- d. If the previous data sets are valid, check the last five runs to see if they are all positive or negative, excluding any zeroes. If they are all positive or negative, then calculate the average difference from the baseline for five sets (25 individual analyses) otherwise proceed to analyze market samples.
  - (1) If the average difference is  $\pm 0.08$  percent protein or less and  $\pm 0.05$  percent oil or less, proceed to analyze market samples.

- (2) If the average difference is greater than  $\pm 0.08$  percent protein or  $\pm 0.05$  percent oil or less, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
  - (a) If the corrected difference is  $\pm 0.08$  percent protein or less or  $\pm 0.05$  percent oil or less, proceed to analyze market samples.
  - (b) If the difference is still greater than  $\pm 0.08$  percent protein or  $\pm 0.05$  percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.

### 3.4 CORN BIAS ADJUSTMENTS

a. General.

- (1) The NIRT instrument may, at times, read either high or low when compared to the SRS baseline values. Systematic deviation from the baseline values greater than the allowable tolerance limits must be corrected before official corn protein, oil, and starch testing services are performed.
- (2) All NIRT instruments are equipped with an "intercept" constant which is used to adjust the instrument's bias with respect to the baseline. If the intercept constant is set properly, the instrument should give results which, on average, are accurate with respect to ISE standard NIRT instruments. Every effort should be made to keep the calculated average bias of all NIRT instruments within  $\pm 0.05$  percent protein,  $\pm 0.06$  percent oil, and  $\pm 0.15$  percent starch at all times.

Adjust the bias of the NIRT calibration by subtracting the calculated average difference between the SRS results and the baseline value from the instrument's INTERCEPT constant for the constituent of interest. This may mean running extra sets of SRS to have two or three valid sets available.

- (3) Prior to official corn protein, oil, and starch testing, perform a bias check and, if necessary, adjust the intercept constant for each constituent.

- (4) Determine instrument accuracy by comparing SRS results to their established baseline values.
  - (5) Only SRS results obtained using procedures identical to market sample procedures should be used in computing bias adjustments.
  - (6) Any change in bias (especially if the bias has been stable for some time) may be an indication of procedural or equipment problems. If in doubt, check for possible problems before making an adjustment.
  - (7) It is permissible to repeat the entire SRS set and discard prior SRS results if the results appear to be in error.
  - (8) Maintain bias records at each location at all times.
- b. Tolerance Levels. In addition to the individual sample difference limit of  $\pm 0.40$  percent protein,  $\pm 0.50$  percent oil, and  $\pm 0.80$  percent starch, four average difference tolerance levels were developed. These tolerance levels allow testing locations to bias to tighter tolerances. The applicable tolerance level is based on the number of valid SRS data sets available.

Presently, the four tolerance levels are set as follows:

- (1) **LEVEL-I ( $\pm 0.15$  percent protein,  $\pm 0.15$  percent oil, and  $\pm 0.35$  percent starch tolerances):**

Apply a LEVEL-I tolerance when evaluating a single valid SRS data set (typically an average of 8 results).

- (2) **LEVEL-II ( $\pm 0.10$  percent protein,  $\pm 0.10$  percent oil, and  $\pm 0.25$  percent starch tolerances):**

Apply a LEVEL-II tolerance when evaluating the average of two valid SRS data sets (typically an average of 16 results) collected within a 2-week period and within a 5°F temperature range.

- (3) **LEVEL-III ( $\pm 0.07$  percent protein,  $\pm 0.07$  percent oil, and  $\pm 0.20$  percent starch tolerances):**

Apply a LEVEL-III tolerance when evaluating the average of three consecutive SRS data sets (typically an average of 24 results) collected within a 2-week period and within a 5°F temperature range.

(4) **LEVEL-IV ( $\pm 0.05$  percent protein,  $\pm 0.06$  percent oil, and  $\pm 0.15$  percent starch tolerances):**

Apply a LEVEL-IV tolerance when evaluating the average of five valid SRS data sets (typically an average of 40 results) that are all positive or negative and were collected within a 2-week period and within a 5°F temperature range. An average difference between a single valid SRS data set and the baseline of zero is neither positive nor negative.

- c. Performing a Bias Check or Adjustment. Previous SRS NIRT results are invalidated after a change in the standard slope, bias adjustment, instrument repair or replacement, or when the recorded temperature has varied by more than  $\pm 5^\circ\text{F}$ .

Perform a bias check when: (1) once a day when the instrument is turned on and warmed up, or after power is restored after a power outage; (2) the instrument's accuracy is questioned; (3) the RH is outside of the acceptable range or RH returns to the acceptable range and a bias adjustment was made while the RH was outside the acceptable range; or (4) the temperature changes by more than  $\pm 5^\circ\text{F}$  from the temperature recorded during the daily check.

**Note: After changing between the 18 millimeter and 30 millimeter sample cell, select the high and low oil SRS samples. Run these samples as a check to verify that the sample cell is installed correctly. The samples should yield oil results similar to the results made prior to changing the sample cell. If not, reinstall the sample cell making sure the area is clear of any obstructions.**

The following procedure outlines the instrument check and evaluation of two kinds of data. These data concern (1) individual differences from the baseline and (2) the data related to the average differences from the baseline. Each is used to determine the relative accuracy of the equipment and the amount of correction (biasing) needed to allow the equipment to duplicate the values of the known SRS baseline values. The instrument check and data evaluation procedure start with "STEP 1" then proceed through a series of "YES" and "NO" responses to questions until being directed to analyze market samples.

**STEP 1:** Mix each SRS thoroughly before analyzing.

- STEP 2:** Calculate the difference between the duplicate analyses for the same sample. Does the duplicate difference for any sample differ by more than  $\pm 0.30$  percent protein, or  $\pm 0.40$  percent oil, or  $\pm 0.90$  percent starch?
- a. If **NO**, proceed to STEP 3.
  - b. If **YES**, reanalyze the sample. Record and use the results from the two analyses closest to each other for the constituent of interest and discard the third result. Proceed to STEP 3.

- STEP 3:** Calculate individual analysis differences between the NIRT and baseline for each constituent. Does any SRS differ by more than  $\pm 0.40$  percent protein, or  $\pm 0.50$  percent oil, or  $\pm 0.80$  percent starch from its baseline value?
- a. If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
  - b. If **YES**, calculate the range of difference between the NIRT and baseline. Is the range greater than 0.50 percent protein, or 0.60 percent oil, or 1.50 percent starch?

Examples for calculating the range difference between two extreme values can be found on page 3-9.

- (1) If **YES**, reanalyze deviating sample(s). Drop from the average any sample that remains more  $\pm 0.40$  percent protein, or  $\pm 0.50$  percent oil, or  $\pm 0.80$  percent starch different from its baseline value and notify your NIRT coordinator. Calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
- (2) If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.

- STEP 4:** Review available data sets. (Data sets older than 2 weeks or intervening bias adjustment, new standard slope, instrument repairs, etc., render SRS data invalid).
- a. Determine whether the average difference between the NIRT and baseline is  $\pm 0.15$  percent protein, or  $\pm 0.15$  percent oil, or  $\pm 0.35$  percent starch.

- (1) If the average difference between the NIRT and baseline is  $\pm 0.15$  percent protein or less, or  $\pm 0.15$  percent oil or less, or  $\pm 0.35$  percent starch or less and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.b.
  - (2) If the average difference between the NIRT and baseline is greater than  $\pm 0.15$  percent protein, or  $\pm 0.15$  percent oil, or  $\pm 0.35$  percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
    - (a) If the corrected difference is  $\pm 0.07$  percent protein or less, or  $\pm 0.07$  percent oil or less, or  $\pm 0.20$  percent starch or less proceed to analyze market samples.
    - (b) If the difference is still greater than  $\pm 0.07$  percent protein, or  $\pm 0.07$  percent oil, or  $\pm 0.20$  percent starch, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- b. If the previous data set is valid, calculate the average difference from the baseline for the two sets (16 individual analyses).
- (1) If the average difference is  $\pm 0.10$  percent protein or less, or  $\pm 0.10$  percent oil or less, or  $\pm 0.25$  percent starch or less and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.c.
  - (2) If the average difference is greater than  $\pm 0.10$  percent protein, or  $\pm 0.10$  percent oil, or  $\pm 0.25$  percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
    - (a) If the corrected difference is  $\pm 0.07$  percent protein or less, or  $\pm 0.07$  percent oil, or  $\pm 0.20$  percent starch proceed to analyze market samples.

- (b) If the difference is still greater than  $\pm 0.07$  percent protein, or  $\pm 0.07$  percent oil, or  $\pm 0.20$  percent starch recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
  - c. If the previous three data sets are valid, calculate the average difference from the baseline for the three sets (24 individual analyses).
    - (1) If the average difference is  $\pm 0.07$  percent protein or less, or  $\pm 0.07$  percent oil or less, or  $\pm 0.20$  percent starch or less and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.d.
    - (2) If the average difference is greater than  $\pm 0.07$  percent protein, or  $\pm 0.07$  percent oil, or  $\pm 0.20$  percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
      - (a) If the corrected difference is  $\pm 0.07$  percent protein or less, or  $\pm 0.07$  percent oil or less, or  $\pm 0.20$  percent starch or less proceed to analyze market samples.
      - (b) If the difference is still greater than  $\pm 0.07$  percent protein, or  $\pm 0.07$  percent oil, or  $\pm 0.20$  percent starch recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
  - d. If the previous data sets are valid, check the last five runs to see if they are all positive or negative, excluding any zeroes. If they are all positive or negative, then calculate the average difference from the baseline for five sets (40 individual analyses) otherwise proceed to analyze market samples.
    - (1) If the average difference is  $\pm 0.05$  percent protein or less, or  $\pm 0.06$  percent oil or less, or  $\pm 0.15$  percent starch or less proceed to analyze market samples.
    - (2) If the average difference is greater than  $\pm 0.05$  percent protein, or  $\pm 0.06$  percent oil, or  $\pm 0.15$  percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.



- (a) If the corrected difference is  $\pm 0.07$  percent protein or less, or  $\pm 0.07$  percent oil or less, or  $\pm 0.20$  percent starch or less proceed to analyze market samples.
- (b) If the difference is still greater than  $\pm 0.07$  percent protein, or  $\pm 0.07$  percent oil, or  $\pm 0.20$  percent starch recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.

### 3.5 INSTRUMENT CHECKOUT

Certain checks and maintenance steps must be performed to verify that the NIRT instruments are functioning properly prior to providing official testing services.

a. Instrument Checkout Schedule.

- (1) Locations providing NIRT testing on a daily basis must complete these checks for each day.
- (2) Locations providing infrequent NIRT testing and/or those that monitor NIRT activities of specified service points must complete these checks before any official NIRT results are provided.
- (3) The instrument checkout procedures outlined below are general. If the instrument does not pass the checkout sequence, official testing shall be suspended until the problem is resolved or corrected. **The first step toward resolution is to repeat the test in question and seek advice from your NIRT coordinator. If the problem cannot be resolved, seek advice from ISE.**

b. Instrument Checkout Procedures.

- (1) Each instrument must have the monochromator checked, the sample cell pathlength standardized (unless you have an Infracore 1229 with a variable sample cell), and use the appropriate standard slope settings for each class of wheat tested and/or soybeans and/or corn tested at the specified service point.

- (2) Analyze the appropriate SRS following official procedures each day or prior to use, as applicable (see 3.5.a).
  - (3) Dust out the sample hopper and path at the end of each day.
  - (4) If more than one instrument is being used at a location, check to make sure the correct calibration disk is being used. Each instrument has its own specific calibration disk labeled with the unit's serial number and the instrument's "O" and "P" constants entered on the disk.
- c. Other Tests. The NIRT instrument performs extensive operational checks on itself whenever it is powered on and during operation. Record any error messages that appear for use in troubleshooting instrument problems.

It is the responsibility of the NIRT coordinator and technicians to alert the supervisor and suspend official testing if the NIRT performance is questionable. **Use the SRS to test instrument accuracy if instrument performance is questioned.** Official testing may resume when acceptable instrument performance is demonstrated.